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Performance Characteristics of Electroless Ni-P-Cu and Ni-P-Cu-Pvp Composite Coatings

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Abstract

In this paper electroless coating on mild steel substrate and testing of the properties of the mild steel after coating were employed. Electroless Ni-P coating and composite coatings were obtained on the mild steel by adding $CuSO_4$ and PVP (Poly vinyl pyrrolidone) .The electroless Ni-P-Cu and Ni-P-Cu-PVP coatings were optimized using trial and error methods. The micro hardness and ultimate tensile strength of composite coatings were higher than that of uncoated samples. SEM and XRD techniques were used for surface morphological and structure of the electroless Ni-P particles and Ni-P-Cu-PVP particles in the coatings. Heat treatment of coated samples were found to improve the mechanical properties of the coatings in comparison with as plated substartes.

Keywords: Corrosion resistance, Hardness, Electroless nickel plating, tensile, SEM.

Introduction

Electroless nickel plating is a process for depositing a nickel alloy from aqueous solutions onto a substrate without the use of electric current. It differs, therefore, from electroplating which depends on an external source of direct current to reduce nickel ions in the electrolyte to nickel metal on the substrate. Electroless nickel plating is a chemical process which reduces nickel ions in solution to nickel metal by chemical reduction. The most common reducing agent used is sodium hypophosphite. Alternatives are sodium borohydride and dimethyl amine borane, but they are used much less frequently. It is estimated that sodium hypophosphite is used in more than 99% of all electroless nickel plating and this publication refers only to the use of this reducing agent. Some of the unique properties of electroless nickel, such as thickness uniformity, hardness, corrosion resistance and magnetic response have resulted in its use in many different industries. In spite of this, not all designers, engineers, metallurgists and others responsible for materials selection are aware of the value of electroless nickel as an engineering or functional coating. However, it is firmly established as a functional coating in the electronics, oil and gas, chemical, aerospace and automotive industries, for instance [1-10]. Hence this process has gained much importance in industrial practice due to its enhanced mechanical properties of the resultant coatings. From the detailed literature survey it is evident that no concrete publication is available for the electroless Ni-P-Cu and Ni-P-Cu-PVP composite coatings and evaluation of its characteristics.

Experimental Procedure

Mild steel was used as substarte material in this study owning to its several industrial practicability. Three types of coatings viz., electroless Ni-P coating, electroless Ni-P-Cu coating and electroless Ni-P-Cu-PVP (Poly vinyl Pyrrolidone) coatings were performed on mild steel specimens of size 20mmx 50mm. The composition of the plating formulation is given as [11] (Nickel sulphate 25 g/L, Sodium hypophosphite ,20 g/L Sodium gluconate 52 g/L, Lactic acid 10 mL/L, Lead nitrate 0.5 mg/L, Sodium lauryl sulphate 0.05 g/L) . The pH of solution has been maintained as 4.7 - 5.2 for maintaining pH small amount (10 to 20ml) of ammonia added to this solution. Temperature of the solution was maintained at 80°C. The Ni-P-Cu coatings were done by adding $CuSO_4$ (0-10 g/l) to electroless Ni-P solution. [4] Ni-P-Cu-PVP coatings were obtained by adding $CuSO_4$ and PVP (0-10 g/l) to this solution. Heat treatment of coated samples was carried out in a muffle furnace at 4000C temperature. Then the mechanical properties such as microhardness by Vickers's hardeness testing, tensile test by Instron tester were measured. SEM and XRD techniques were employed to understand the surface morphology and structure of the coatings.

Micro hardness measurements

Micro hardness measurements [8] for electroless coated as plated and heat treated samples (20x 50 x 2 mm3) were measured using Vicker's harness tester with a load of 100 g. A diamond shaped indentation was created on 6 parts of each as plated as well as heat treated samples.

From the average of these readings, micro hardness was calculated from the diagonal of indendation on Vicker's scale using the formula.

V.H.N = (1854 x load) / d^2, Where d = diagonal of the indenter

Tensile Test

Tensile specimens are made according to ASTM E8 [14] standard and this test determines important mechanical properties such as yield strength, ultimate tensile strength, elongation, and reduction of area. E8 tensile tests determine the ductility and strength of metal when the materials undergo uniaxial tensile stresses. Tensile test of as plated as well as heat treated samples were done in the INSTRON testing machine.

Surface Morphological Studies

SEM and XRD techniques [10][5] were used to understand the surface morphology and structure of the electroless coatings. All as plated as well as heat treated samples with size 40X10mm and 10X10mm were used for the XRD test SEM - EDX analysis.

Surface roughness

Surface roughness [14] of the coated and uncoated samples were carried out using Mahr-surface roughness tester.

Result and Discussion

Micro hardness measurements

Table 1: Microhardness Values of Coated Samples

Type of coating	As Plated(V H N)	Heat Treated(V H N)
Electroless Ni-P coating	495 V H N	980 V H N
Electroless Ni-P-Cu coating	582 V H N	1063 V H N
Electroless Ni-P-Cu -PVP coating	690 V H N	1100 V H N
Mild steel	180 V H N	--

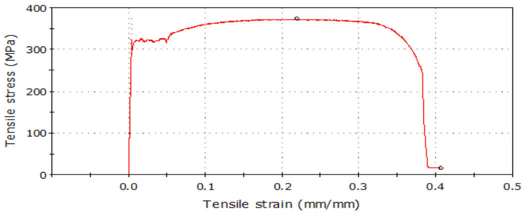
Microhardness of electroless coated samples was found increased. Electroless Ni-P coated samples have higher % micro hardness than the uncoated mild steel, and after heat treatment of, hardness values are getting approximately doubled. It was observed that about 5g l/l of each Cu and PVP are found sufficient for bath loading to develop composite coatings on mild steel. .This is due to the fact that beyond this concentration, agglomeration of particles has started. g It has been noticed that hardrdness value of electroless Ni-P-Cu coatings are higher than that of Ni-P due to addition Cu powder both in the as plated as well as heat treated samples. Also the addition of PVP powders enhance the micro hardness by forming intermetaalic phases which is further facilitated by precipitation hardening process after heat treatment at 400^0C on the coatings. The results are presented in table 1.

Tensile test

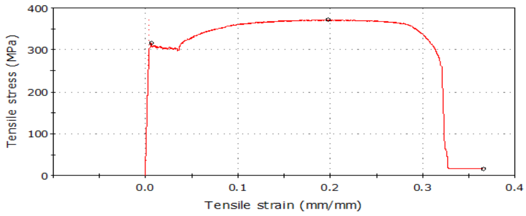
Table 2: Results of Tensile Tests For Electroless Composite Coatings

Type of coating	Tensile strain at maximum load (%)	Tensile stress at break (standard) (MPa)	Maximum load (N)	UTS (GPa)	% Elong. at Tensile Strength at Non-proportional Elongation (Standard) (%)
Uncoated Mild steel	21.93265	15.56	4658.48446	0.361	20.99973
Electroless Ni-P coated as plated	19.74332	16.16	5121.71984	0.372	19.11362
Electroless Ni-P coated heat treated	19.94454	21.67	5073.89307	0.373	19.88650
Electroless Ni-P-Cu coated as plated	20.84679	35.82	5031.68106	0.398	20.24755
Electroless Ni-P-Cu coated Heat treated	21.33213	36.86	5041.18204	0.403	21.64930

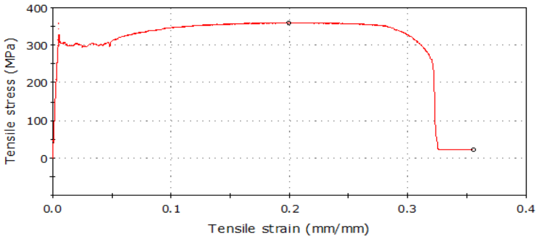
Electroless Ni-P-Cu-PVP coated as plated	20.85515	17.78	4670.01200	0.377	20.49117
Electroless Ni-P-Cu-PVP coated heat treated	21.98962	23.29	4972.95856	0.395	20.67209



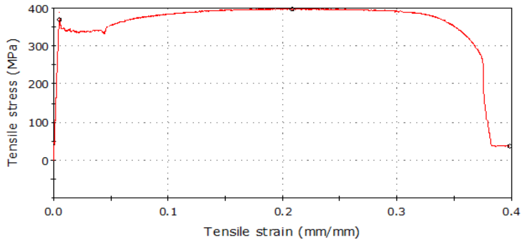
Uncoated mild steel



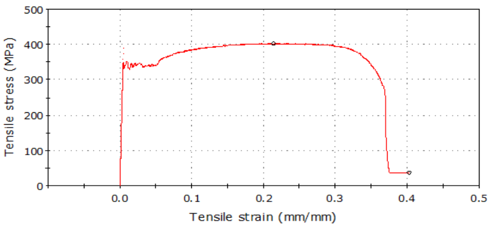
Electroless Ni-P coated as plated



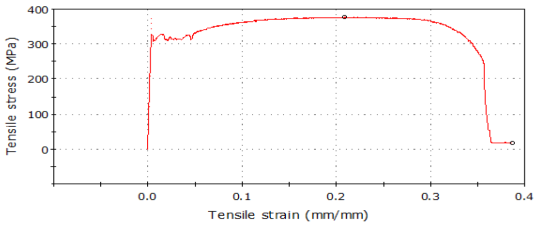
Electroless Ni-P Heat treated



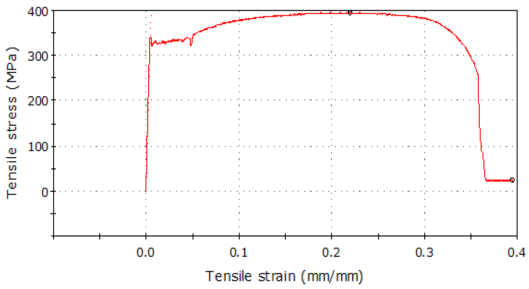
Electroless Ni-P-Cu as plated



Electoless Ni-P-Cu Heat treated



Electroless Ni-P-Cu-PVP as plated



Electroless Ni-P-Cu-PVP heat treated

Figure 1: Stress -strain curves of uncoated and electroless Ni-P-Cu and Ni-P-Cu-PVP coated mild steel

The tensile strength results of the coatings are given in table 2 and in fig 1.It was noted that up to the elastic limit there is not much differences in tensile strain values for both coated and uncoated mild steel. There is some differences after the yield stress, and the tensile stress is were visualized. It is found that tensile stress is high for the coatings .It has been also observed that Ni-P-Cu coatings are showing higher ultimate tensile strength both as plated as well as heat treated samples than electroless Ni-P and Ni-P-Cu-PVP. This is due to the existence of copper particles in the coatings which is a ductile metal and having elongation property. During coating, the incorporation of PVP particles into nickel matrix impeded the co deposition of copper.

Surface roughness

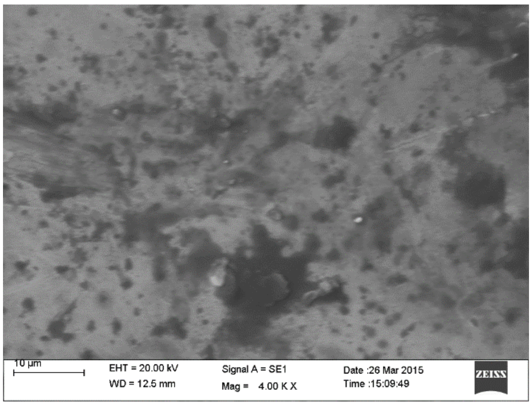
Table 3: Surface roughness values of electroless composite coatings

	Ra(µm)			
Type of coating	1	2	3	Average
Uncoated Mild steel	1.5721	1.0244	1.2548	1.2837
Electroless Ni-P coated as plated	0.9752	0.8849	0.8256	0.8952
Electroless Ni-P coated heat treated	0.5173	0.3612	0.5561	0.4782
Electroless Ni-P-Cu coated as plated	2.0003	1.2270	2.0753	1.7675
Electroless Ni-P-Cu coated Heat treated	1.3508	1.1399	1.1432	1.2113
Electroless Ni-P-Cu-PVP coated as plated	0.7313	1.2512	1.1250	1.0358
Electroless Ni-P-Cu-PVP coated heat treated	1.0955	1.1423	1.0578	1.0985

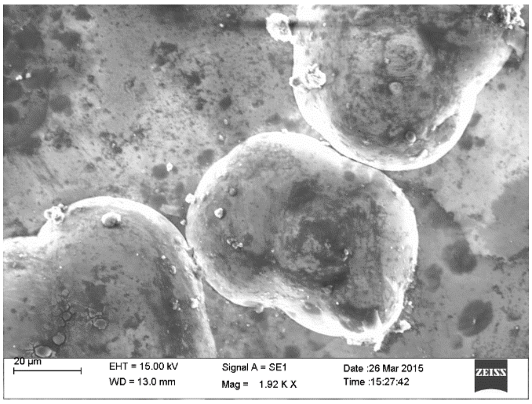
Surface roughness of coated samples are appeared as lesser than the uncoated mild steel. Also electroless Ni-P-Cu-PVP composite coatings are possessing lower roughness both in the as plated as well as heat treated conditions due to the incorporation of soft polymer particles i.e., PVP. The results are presented in table 3.

Surface morphological studies

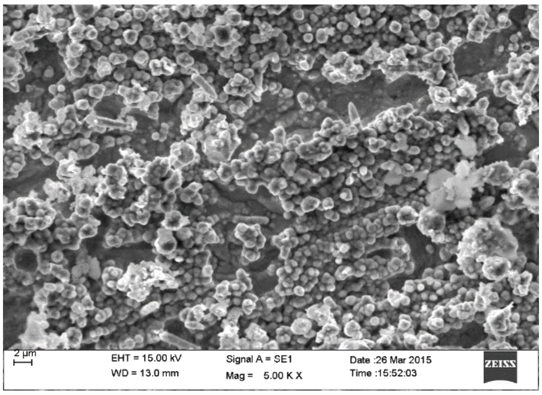
SEM analysis images shown in figure 2



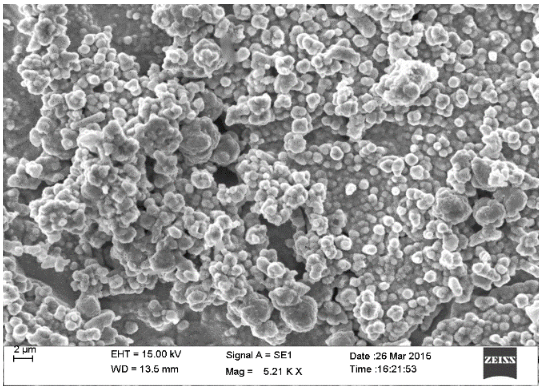
Electroless Ni-P coated as plated



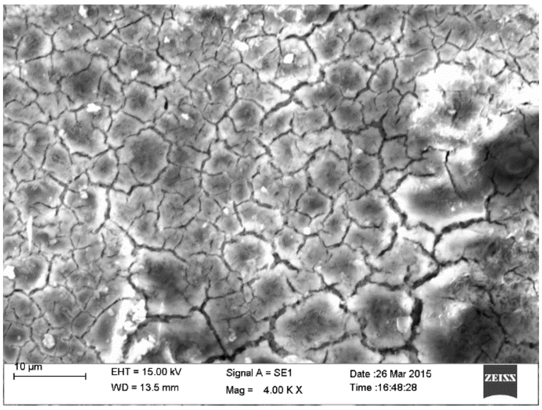
Electroless Ni-P Heat treated



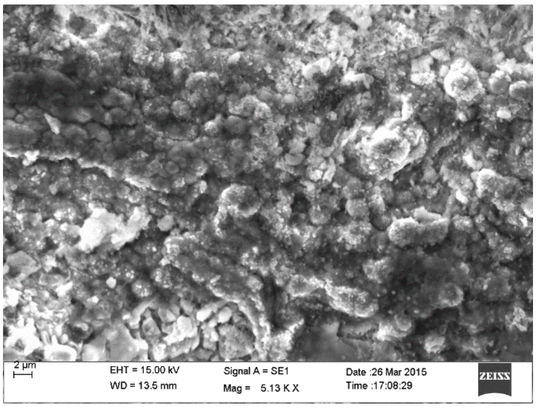
Electroless Ni-P-Cu as plated



Electoless Ni-P-Cu Heat treated



Electroless Ni-P-Cu-PVP as plated



Electroless Ni-P-Cu-PVP heat treated

Figure 2:

SEM images of electroless Ni-P coating in the as plated samples indicate the existense of pores and pits in the surface there is no nodules are found on the surface. After heat treatment Electroless Ni-P coating are showing bigger sphericle particles , like pumpkin structure on the surface. The images of Electroless Ni-P-Cu indicated that the formation of spherical particles island due to the presence of intermetalic phases, viz., Ni_2Cu_5. After heat treatment the aggregation of Ni-Cu particles are more due to the precipitation hardening of coatings. Electroless Ni-P-Cu-PVP coating are showing segmented shapes and showing pites between the segments. Heat treatment of this coating have improved the both Copper and PVP particles crowding on the coatings by nullifying pits and dendrites through the formation of a densed structure.

Conclusion

- Micro Hardness of composite coatings are higher than the uncoated samples both in the as plated as well as heat treated samples.
- The electroless Ni-p-Cu and Ni-P-Cu-PVP coatings were found to increase the ultimate tensile strength and load carrying capacity of mild steel.
- Surface roughness of coated mild steel are lesser than that of uncoated mild steel
- The formation of island of spherical particles and its aggregation for composite coatings have clearly confirmed the existence of copper and PVP in the electroless Ni-P matrix.

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